

Fast purification and characterisation of semiochemical compounds from essential oils of *Matricaria chamomilla* L. and *Nepeta cataria* L. using column chromatography fractionation and Ultra Fast GC analysis

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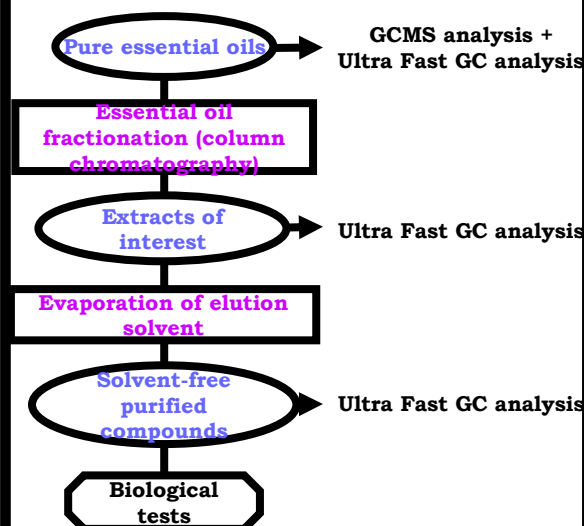
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Introduction

The present study describes both, a fast, simple and reproducible original method for obtaining purified essential oil extracts of interest in chemical ecology, and fast GC analysis for their characterisation. Biological tests are currently conducted on aphid's predators (*Episyrphus balteatus*) and parasitoids (*Aphidius*

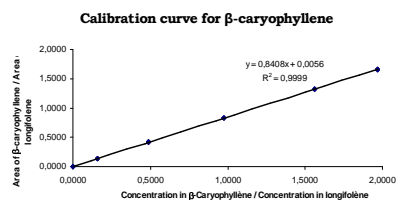
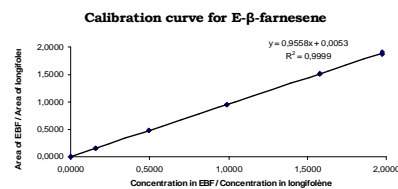
Experimental

MATERIAL AND METHODS



Ultra Fast GC analyses validation

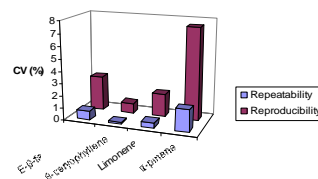
Examples of calibration curves for E-β-farnesene and β-caryophyllene with longifolene as internal standard



LOD and LOQ (in ng.ml⁻¹)

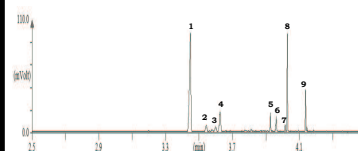
| | E-β-farnesene | β-caryophyllene | Limonene | α-pinene |
|-----|---------------|-----------------|----------|----------|
| LOD | 2.40 | 0.65 | 1.39 | 1.74 |
| LOQ | 4.79 | 1.30 | 2.78 | 3.48 |

Example of coefficient of variation at 0.48 μg/μl (repeatability and reproducibility)



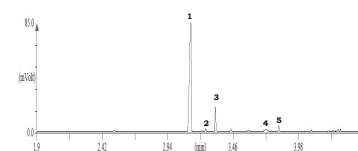
Characterisation of essential oils by Ultra Fast GC

Matricaria chamomilla



| Major compounds | Composition | Kovats indices |
|--------------------------|-------------|----------------|
| 1 E-β-farnesene | 42.6 % | 1456 |
| 2 Germacrene D | 2.9 % | 1478 |
| 3 Bicyclogermacrene | 2.0 % | 1494 |
| 4 (E,E)-α-farnesene | 8.3 % | 1506 |
| 5 α-bisabolol oxide B | 4.4 % | 1649 |
| 6 α-bisabolone oxide A | 4.5 % | 1673 |
| 7 Chamazulene | 1.2 % | 1715 |
| 8 α-bisabolol oxide A | 21.2 % | 1735 |
| 9 Cis-en-in-dicycloether | 5.9 % | 1802 |

Nepeta cataria



| Major compounds | Composition | Kovats indices |
|--------------------------------------|-------------|----------------|
| 1 (Z,E)-nepetalactone ^(*) | 73.3 % | 1353 |
| 2 (E,Z)-nepetalactone | 1.1 % | 1377 |
| 3 β-caryophyllene | 9.7 % | 1415 |
| 4 unknown | 7.8 % | / |
| 5 β-caryophyllene oxide | 1.8 % | 1579 |

(*) Stereoisomery confirmed by ¹H and ¹³C NMR

Essential oils fractionations

Purification of semiochemical compounds

Matricaria chamomilla

| Compounds | F2 | F3 | F4 |
|---------------------|--------|--------|--------|
| Sum of monoterpenes | 11.5 % | 1.3 % | / |
| E-β-farnesene | 75.3 % | 84.0 % | 77.1 % |
| Germacrene D | 1.4 % | 1.4 % | 1.6 % |
| Bicyclogermacrene | 3.9 % | 1.4 % | / |
| (E,E)-α-farnesene | 7.8 % | 11.9 % | 21.3 % |

Nepeta cataria

| Compounds | F2 | F5 |
|---------------------|--------|--------|
| Z,E-nepetalactone | / | 98.3 % |
| β-caryophyllene | 80.1 % | / |
| Caryophyllene oxide | / | 1.7 % |
| Other compounds | 19.9 % | / |

Conclusions

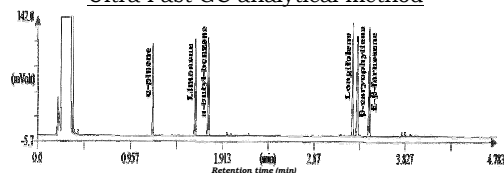
- Development and validation of the method according to ISO 5725
- Low detection and quantification limits
- Enrichment of fractions in compounds of interest
- Fastness, repeatability, reproducibility

Acknowledgment

The authors gratefully acknowledge Prof. M. Luhmer (Département de chimie, Laboratoire de RMN haute résolution, ULB, Belgium) for NMR analyses.

CHROMATOGRAPHIC CONDITIONS

Ultra Fast GC analytical method



Oven :
Initial T° : 40°C; 0.10 min
Ramp 1 : 30°C/min → 95°C
Ramp 2 : 35°C/min → 155°C
Ramp 3 : 200°C/min → 280°C; 0.5 min
Oven run time : 4.78 min

Ultra Fast Module : Ph5; 0.1 μm film thickness, 5m x 0.1mm I.D.
Carrier gas : He; 0.5 ml/min
Split ratio : 1:100